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DEVELOPING HIGH-TEMPERATURE WATER-SOLUBLE COATINGS FOR RECONFIGURABLE TOOLING MATERIALS

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High-Temperature water-soluble coatings are under development for the reformable state-change tooling materials of 2Phase Technologies' rapid, low-cost reconfigurable tooling systems (RTS). Such coatings can provide surface finish, impermeability, and improved structural properties for these materials. This paper describes the development of such coatings, including the investigation of chemical modification and mechanical reinforcement as well as the suitability of the coatings in elevated-temperature use. The coatings developed are based upon the same binder used for the tooling materials in the current commercial 2Phase reconfigurable tooling systems. Coatings solution properties, including solution density, pH values, and wet ability, were measured at different binder compositions. In addition, optical microscopy was employed to evaluate the coating texture. The paper describes the high-quality coatings obtained and the approach to further development.

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ABSTRACT

High-temperature water-soluble coatings are under development for the reformable state-change tooling materials of 2Phase Technologies' rapid, low-cost reconfigurable tooling systems (RTS). Such coatings can provide surface finish, impermeability, and improved structural properties for these materials. This paper describes the development of such coatings, including the investigation of chemical modification and mechanical reinforcement as well as the suitability of the coatings in elevated-temperature use. The coatings developed are based upon the same binder used for the tooling materials in the current commercial 2Phase reconfigurable tooling systems. Coating solution properties, including solution density, pH values, and wettability, were measured at different binder compositions,. In addition, optical microscopy was employed to evaluate the coating texture. The paper describes the high-quality coatings obtained and the approach to further development.

KEY WORDS: Coatings, Affordability Technology, Tooling Technology

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1. INTRODUCTION

Reformable tooling systems are under development to permit rapid tooling modification and reconfiguration for rapid, low-cost open- or close-molding of composite materials and liquid resins. This technology relies upon 2Phase Technologies' patented state-change materials, which are liquid-particulate mixtures that can be changed from a formable (liquid-like) state to a force-resisting (solid) state, and then back to a formable state without change in volume.[1,2] Once the desired tooling configuration is captured the water-based inorganic binder in these materials allows the tool to be "cured" at moderate temperature to produce semi-permanent tooling, and yet this cure can be reversed and the material reformed at any time by reinjection of the binder solution. This permits reconfiguration of the tool to accommodate design changes or for use of the tooling to make a completely different part.

Due to the porous surface of the tooling materials after cure, some type of liquid-resin impermeable layer or coating must be used to allow composite lay-up without penetration of the tooling material by the resin matrix. Thin elastomeric membranes have been the primary solution to date, but a desirable alternative is the development of a coating that, like the tooling material itself, will be reformable while still conferring resin impermeability. This paper describes the ongoing development of a water-soluble coating for the reformable state-change tooling materials used in a reconfigurable tooling system (RTS) and reports on the high-quality coatings developed to date.

Combining resin impermeability with the fact that coated 2Phase tooling materials will be used up to 400^{o}C (750^{o}F), coating development must take into account the following:

1. Solubility of the coating must be inhibited or calibrated to avoid depletion of the coating during "wet" cycles of the tooling material;
2. The coating must sustain precision tolerances through tool- and part-production cycles;
3. The coating must have high temperature resistance, a strong bond to the hardened particle/binder mix, and chemical and dimensional stability even after high temperature cycling;
4. The coefficient of thermal expansion (CTE) must match the substrate material in order to avoid delamination or cracking and to maintain dimensional tolerances;
5. The solubility after high-temperature exposure must be maintained so that the coating can be easily (preferably automatically) stripped from a fabricated shape;
6. The coating needs sufficient strength to withstand the pressure applied when fabricating composites;
7. The mechanical strength of the coating must be sufficient to avoid rupture during gelation and cure;

8. The coating must be impermeable to resin up to and at the working temperature; and
9. The coating needs to demold with a smooth surface and maintain smoothness during composite processing.

2. COATING DEVELOPMENT CONCERNS AND PROCEDURE

The same water-soluble binder already in use in the 2Phase reconfigurable tooling materials was used to develop coatings for the porous surface of the tooling materials.

2.1 Coating development concerns

2.1.1 Trapped water and trapped air in the coating The first factors that had to be considered in creating such a coating are the possibilities of water or air trapped in the coating.

There are three ways in which water can be trapped in the water-soluble binder: as free water, as secondary chemical-bonded water, or as primary chemical-bonded water. Most of the free water will evaporate or be removed by vacuum at room temperature; secondary chemical-bonded water can be removed by elevating the temperature; primary chemical-bonded water will be removed or remain depending upon the hydration state and will be released above some certain elevated temperature. As the temperature is increased during tooling material hardening or composite curing, any water still trapped in the coating may bubble and ruin the surface smoothness.

Similarly, air can be trapped in the binder, and any trapped air will also bubble as temperature is increased, ruining the surface smoothness. The problems with both trapped water and trapped air will become more severe the thicker the coating.

2.1.2 Chemical modifiers Two types of chemical modifiers were introduced. Chemical modifier I was a defoamer, which was used to reduce the amount of trapped air and decrease the generated bubble size. Chemical modifier II, a surfactant, was used to modify the wettability on a substrate as well as to shift the dehydration temperature.

2.1.3 Glass microspheres Glass microspheres improve the coating strength by mechanically reinforcing the coating. The average diameter of the glass microspheres used in the coatings was about 50 μm, which is much smaller than is used in the reconfigurable tooling materials themselves.

2.1.4 Gelling agent Gelling agents were introduced to retain moisture temporarily, make the coating more flexible and provide a barrier to inhibit coating dissolution. Both alcohol and acetate gelling agents were used.

2.1.5 Substrate 2Phase state-change materials were used as the substrate for all coating studies described in this paper. It should be noted that the water-soluble coating

was developed primarily for use on 2Phase tooling materials but could also be used in other coating applications.

2.2 Coating development procedure

2.2.1 Coating formula preparation First, assemble the 2Phase binder solution, chemical modifier I, chemical modifier II and glass microspheres in the proportions 100:1:1:50 by volume. Second, combine the coating binder, chemical modifier I and chemical modifier II and mix for 5 minutes by mixer at room temperature. Third, add the glass microspheres and mix for an additional ten minutes.

2.2.2 Mold preparation First, clean the mold with water to remove any inorganic dust on the mold surface, then clean with alcohol to remove any organic dust. Second, spray with a mold-release agent and dry the mold in a 65^{o}C (150^{o}F) furnace for 3 minutes.

2.2.3 Coating procedures Three coating methods were investigated:

2.2.3.1 Brush-on method, in which the coating is applied directly to a tool by brushing thin layers, with air drying in between, onto the finished, cured state-change material and then is post-cured to a hardened state;

2.2.3.2 Spray-on method without gelling agent, in which the coating is applied by spraying back and forth onto the finished, cured state-change material or into a mold that will be filled with state-change material. The spray passes continue until a 0.5-mm-thick coating is developed, and then the coating is air dried and subsequently post-cured to a hardened state;

2.2.3.3 Spraying method with gelling agent, in which the coating mixture is applied as in the previous method and then a gelling agent, which can be alcohol or acetate, is sprayed in a single sprayer pass onto the coating layer, followed by air drying and post-curing the combination coating to a hardened state.

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3. MATERIALS PROPERTIES

3.1 **Density and solid content** Due to concerns about alkaline corrosion, pH control is necessary to extend the glass microsphere service life. Changes in binder concentrations in the coating solution change both the density and the pH of the coating solution. Figure 1 shows the relationship between solution density and both binder solid content and pH. Solution density was measured by a hydrometer. Based on measured density and the linear relationship between density and solid content, the solid content can be calculated by a curve fitting equation ($A=1.0158\times\rho-1.0351$, where A: solid content, ρ: solution density).

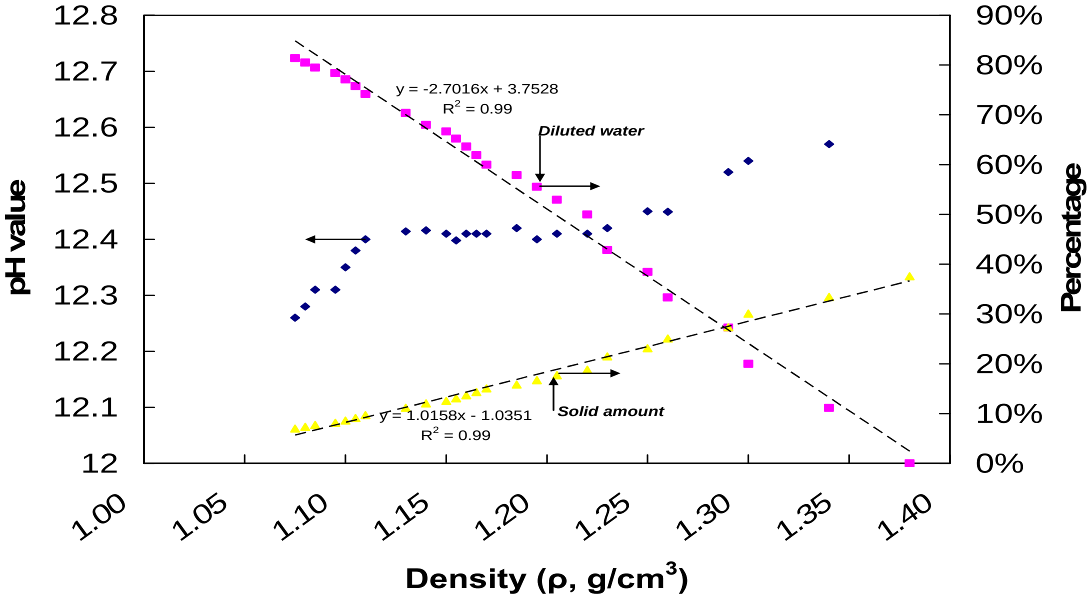


Figure 1. Solution density as a function of pH and increased binder concentration solids in 2Phase coating binder solution

3.2 **Wettability** A CAM 100 compact contact angle meter was employed to investigate the binder surface properties. Figure 2a shows the software interface and Figure 2b shows the wettability of different binder concentrations. The contact angle is found to decrease as binder concentration increases, indicating better wetting by the coating solution.

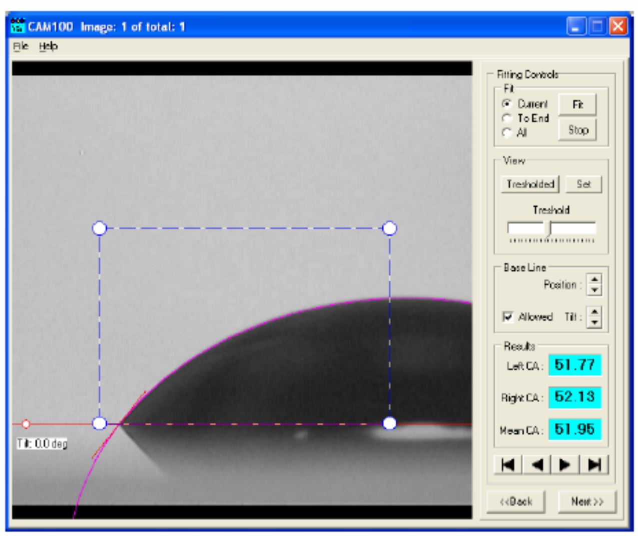


Figure 2a. Contact angle measurement software interface

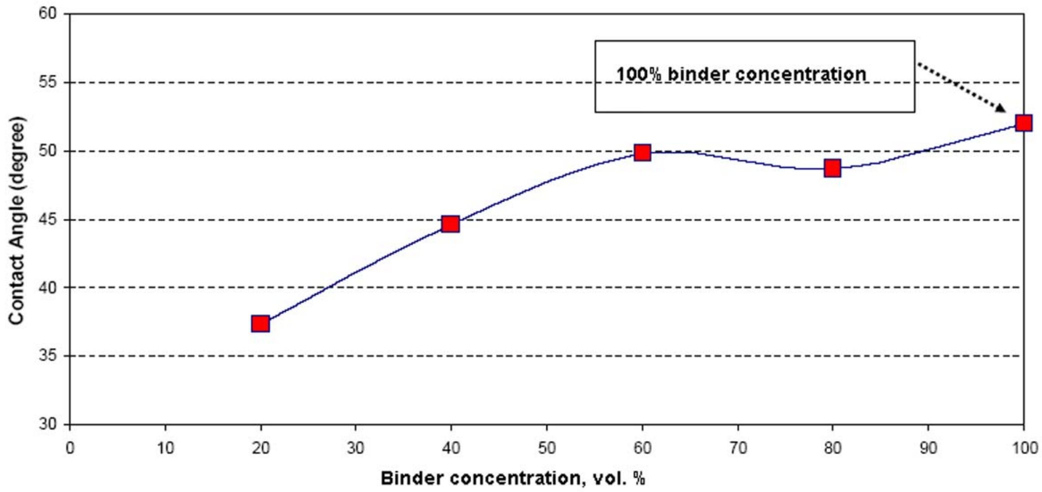


Figure 2b. Binder wettability at different binder concentrations.

4. RESULTS

4.1 Coated tools produced using three coating methods The results of the three coating procedures are shown in Figures 3, 4, and 5.

Figure 3 shows a tool coated using the brush-on method. The resulting coating is non-uniform and shows significant defects.

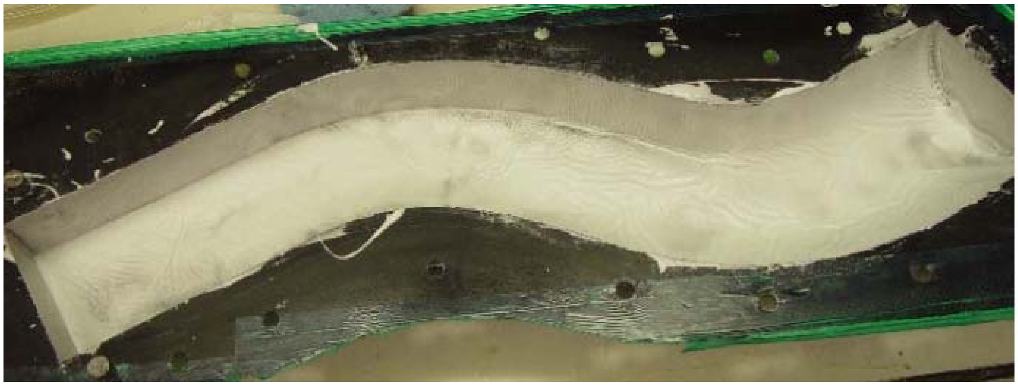


Figure 3: Coating applied to tooling by hand brushing

Figure 4 illustrates how the two spray-on methods were used to apply coatings to the tooling by applying them to a mold in which the tooling is to be formed. Figure 4a shows a mold that has been coated using the spray-on method without gelling agent, while Figure 4b shows a similar mold coated using the spray-on method with gelling agent.

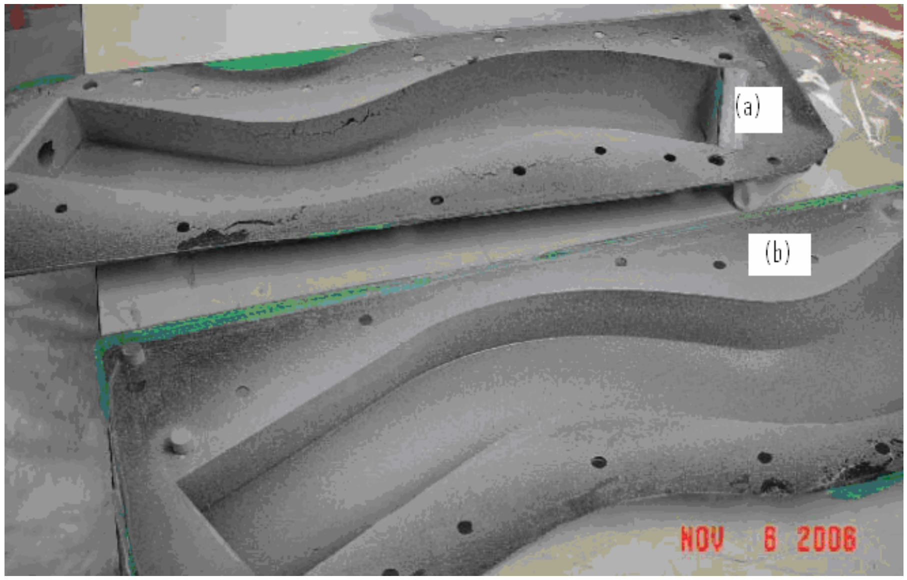


Figure 4. Coating applied to an inner mold by spray-on method: (a) spray-on and air-dry coating without alcohol gelling agent and (b) spray-on and air-dry coating with alcohol gelling agent

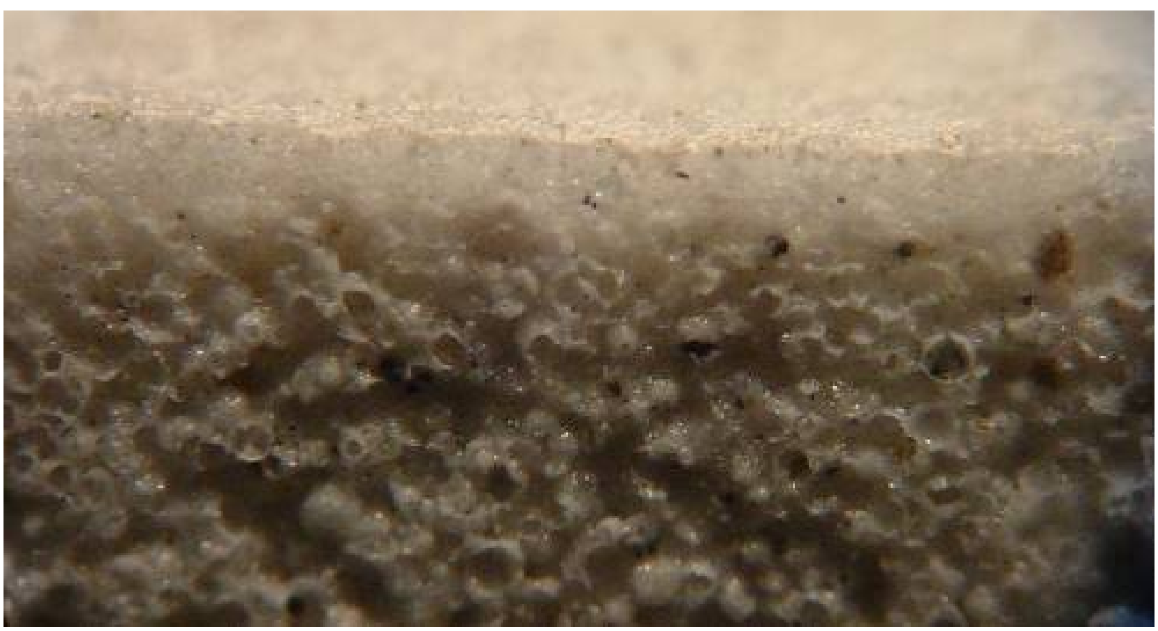
After the coating was applied to the inner mold, a slurry of the liquid-like tooling material was pumped in at one end of the mold while excess liquid was drained at the other end. The tool was then cured at 177^{o}C (350^{o}F) and 550 MPa (80 ksi) vacuum for six hours.

As shown in Figure 5a, the spray-on method with a gelling agent applied to the tooling mold surface generated a very smooth, glass-like coating surface. The gelling agent produces a polymerization reaction that changes the coating into a gel-like substance and reduces the solubility of the coating during the slurry-filling step. For the spray-on coating applied without the gelling agent, however, the coating material dissolved in the tooling material slurry during processing, leaving a rough, permeable surface as shown in Figure 5b(1). The brush-on method, as shown in Figure 5b(2), does not give a uniform coating on the outer surface. The spray-on method with gelling agent generated the best surface: a glass-like, smooth surface that is an exact match to the mold surface.

(a) (b)

Figure 5: Trapped tooling with coatings: (a) shining surface resulting from tooling with spray-on coating method with gelling agent, and (b) comparison of three finished tools: (1) tool with spray-on coating without gelling agent, (2) tool with brush-on coating and (3) tooling with spray-on coating with gelling agent.

4.2 Optical microscopy Figure 6, taken by an optical microscope, shows a tooling surface with a coating produced by spraying-on with an alcohol gelling agent. The micrograph reveals that the coating is smooth, translucent and pinhole-free and is about 400 μm thick.



500 μm _______

Figure 6: Optical micrograph of coating produced by spray-on with gelling agent on a tooling surface of 2Phase Technologies' reconfigurable tooling material.

4.3 Coating suitability for high-temperature usage To evaluate the suitability of the coatings for high-temperature use, portions of tooling specimens with coatings produced by the spray-on method with gelling agent were baked for six hours at 400°C (750°F), as shown in Figure 7. At this high temperature the coating changes to a whiter color but remains smooth and glass-like.

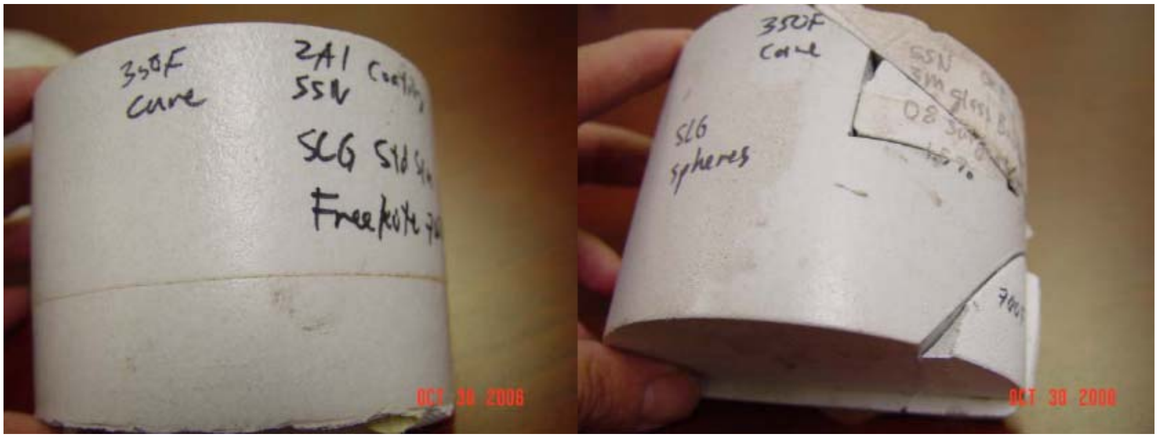


Figure 7. High temperature evaluation of 2Phase spray-on coating with gelling agent. Specimen on right was cured at 177°C (350°F) while sections of the specimen on the right, as marked, were removed and cured at 400°C (750°F).

The whitening is due to foaming in the binder, but chemical modification by chemical additives and mechanical reinforcement by glass microspheres can been used to decrease the foaming bubble size to a microscopic scale.

5. CONCLUSIONS AND FUTURE WORK

A high-temperature-capable, high-quality, water-soluble coating was successfully developed. Such coatings are of particular importance to provide surface finish, impermeability, and improved structural properties for reconfigurable tooling materials. Coating solution properties, including solution density, pH values, and wettability, were measured at different binder concentrations. Three different techniques for producing coatings were evaluated. Optical microscopy was employed to evaluate the resulting coatings. A spray-on technique using a gelling agent was found to produce a high-quality, pin-hole-free, glass-like coating surface.

Future work will include further investigation and development of coating formulations for elevated-temperature service, and development of coating techniques for production-level tooling.

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